



Real-Time, Nanoscale Failure Analysis Achieved

Experimental Technique Allows New Insights into Material Deformation

A research team led by Eric Stach and J.W. Morris has observed for the first time, *in-situ* and in real time, the nanoscale processes that cause metals to “plastically deform.” These experiments, which involved a test that measures hardness, were made possible by the development of a new tool, an “*in-situ* nanoindenter,” at the National Center for Electron Microscopy.

The hardness of a material is defined as its resistance to irreversible or “plastic” deformation. Since the early part of the 20th century, this has been measured quantitatively by forcing an indenter against the sample and relating the size (~10 nm) of the impression made to the force applied. Over the last two decades, nanoindentation techniques have evolved to extend testing to smaller and smaller samples, such as thin films and multilayer structures. In this work, a small diamond tip (50~100 nm radius) is used as the indenter and the force required to produce a certain displacement (the so-called load-displacement curve) is used to quantify the material’s hardness.

At small forces, during the initial stages of deformation, the response of a material is elastic or recoverable. As the force is increased, a point is reached when the deformation becomes plastic. It is known that crystalline defects known as dislocations are formed when a material is plastically deformed. Despite extensive study, however, the mechanisms by which these dislocations are nucleated at the onset of plastic deformation (elastic instability) has remained elusive.

The MSD team addressed this challenge by developing the “*in-situ* nanoindenter” which allows nanoindentation tests inside the sample chamber of a transmission electron microscope at the National Center for Electron Microscopy. They overcame several technical hurdles including the nanoscale positioning of a sharp diamond tip onto an electron transparent sample and the calibration of the load-displacement behavior of the instrument. With this new tool, real-time, atomic-scale images of the material under test can be generated at any point on the load-displacement curve.

The team performed their first experiments on single grains of aluminum. In the data shown in the figure, the onset of plastic deformation is clearly observed when the indenter is forced more than 10 nm into the grain. Analysis of the TEM images revealed that just at the point that elastic instability is reached, “geometrically necessary” dislocations begin to form in the grain (these “geometrically necessary” dislocations occupy less volume than the unperturbed material and thus allow the volume of the indenter to be accommodated). As the indentation proceeds, increasing numbers of these dislocations are introduced into the aluminum grain, thereby causing permanent, plastic, deformation.

Continuing studies of nanoindentation will include experiments to systematically determine the dislocation mechanisms responsible for the observation that as films become thinner their hardness increases. Additionally, because of its ability to correlate load – displacement behavior with nanoscale imaging of materials, the new technique will be exploited to understand the mechanisms of elastic instability in new classes of hard materials, such as carbonitrides (these materials are, in general, only available in thin film form and therefore their hardness can only be measured by nanoindentation).

Eric A. Stach (510) 486-4634, National Center for Electron Microscopy, and J. William Morris, Jr. (510) 486-6462 Materials Science Division (510) 486-4755, E.O. Lawrence Berkeley National Laboratory

E. A. Stach, T. Freeman, A. M. Minor, D. K. Owen, J. Cumings, M. A. Wall, T. Chraska, R. Hull, J.W. Morris, Jr., A. Zettl and U. Dahmen; *Microscopy and Microanalysis*, 7(6), 507-517, 2001

A.M. Minor, J.W. Morris, Jr. and E.A. Stach, *Appl. Phys. Lett.* 79 (11), 1625, 2001.